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EMISSION OF VOLATILE ORGANIC COMPOUNDS (VOC) FROM WATERBORNE LACQUERS WITH DIFFERENT CONTENT OF SOLIDS

This paper presents the results of investigations on the emission of volatile organic compounds (VOC) from oak wood surfaces finished with acrylic waterborne lacquers. The lacquers selected for the experiments were characterised by a different content of solids, fluctuating between 32 and 38%. The volatile organic compounds were analysed using a gas chromatography technique combined with mass spectrometry and thermal desorption (GC/MS/TD). Tenax TA was employed as an adsorption medium. The obtained results indicate that volatile organic compound emissions from the experimental waterborne lacquers varied widely from 388 to 1794 $\mu\text{g}/\text{m}^3$. The most important constituents of emissions included compounds that belonged mainly to glycol ethers as well as aliphatic and aromatic hydrocarbons.

Keywords: waterborne lacquers, volatile organic compounds (VOC), gas chromatography with mass spectrometry and thermal desorption (GC/MS/TD)

Introduction

Surface finishing of wood and wood-based materials with various lacquers has been applied for many years now. Among other things, this kind of finishing is to used in order to give the above-mentioned products the required decorative-aesthetic appearance, to protect them against the destructive influence of external factors, and to ensure their long-term service life. Lacquer application, depending on the type of applied lacquers and the techniques used to apply them, may have a different impact on the natural environment and the microclimate of production halls. Major environmental hazards resulting from lacquer application are connected with organic solvents, whose main task is to give lacquers specific application properties. Most organic solvents are qualified as toxic substances, dangerous due to their volatility as well as ability to dissolve in fats.

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Their properties are such that organic substances emitted from solvents may be absorbed by the skin, mucous membranes or the gastrointestinal tract and cause headaches, dizziness, nausea, severe poisoning and even have depressing or narcotic effects.

The current legislation concerning limitations of air pollution obliges manufacturers to produce lacquer articles which are characterised by reduced concentrations of organic solvents. Among the most important, environmentally-friendly lacquering systems meeting current legislative requirements are products based on waterborne binders. Until recently, it seemed that waterborne lacquers would never threaten solvent products. In the case of the wood industry, the application of waterborne lacquers was frequently connected with a number of different problems, e.g. formation of milky, non-transparent coatings, so-called orange-peel effect, long drying periods or improper wood structure exposure [Lurka 2007]. Once the above-mentioned drawbacks were eliminated, a growing interest in this group of articles has been observed.

At present, the manufacturers of lacquers offer a wide range of different products, especially of waterborne articles, of various properties and of different chemical composition intended for wood and wood-based materials. Waterborne materials manufactured on the basis of acrylic dispersions are among quite popular and frequently-employed lacquers. Some of their numerous advantages which deserve a mention are: resistance to atmospheric factors, good adhesion, coating colour stability, resistance to water and to UV radiation.

It is widely believed that waterborne lacquers are completely safe for the health. Unfortunately, this opinion is incorrect. These products contain resins, pigments, fillers, as well as many other essential aiding agents that are not entirely neutral for the environment. In addition, they also contain certain quantities (5 to 15%) of organic solvents [Asendorf 1996; Proszyk 2007] that are added, inter alia, to reduce a minimum temperature of film formation, facilitate the coalescence of dispersion particles, obtain good fluidity, regulate drying time, and improve rheological properties. Nevertheless, solvents used in waterborne materials are less noxious, because most frequently they belong to the group of glycols, polyglycols, higher alcohols, etherglycols and their esters [Kuczyńska 2005].

Experiments carried out by research centres proved that the application of waterborne lacquers made it possible to considerably reduce the use of harmful emissions in comparison with materials containing solvents [Scheithauer, Aehlig 1995; Salthammer 1997; Dziewanowska-Pudliszak 2007].

Nonetheless, in this group of waterborne lacquers as well, it is necessary to compare their impact on indoor air quality and indicate systems that are the safest both for the natural environment and the health of users. The aim of the investigations carried out was to recognise the effect on the environment of waterborne materials used in the wood industry which are manufactured on the basis of acrylic dispersions and contain different quantities of solids.

Materials and methods

The experiments were conducted on three selected, one-component and colourless lacquers based on waterborne acrylic binders intended for the surface finishing of furniture as well as of other wood materials used indoors. Detailed information about the lacquers employed in the described investigations are presented in table 1. One important selection criteria of the experimental lacquer products was the content of solids, which ranged from 32 to 38%.

Table 1. Properties of applied lacquers (on the basis of the manufacturers' data)
Tabela 1. Parametry techniczne wytypowanych do badań wyrobów lakierniczych (na podstawie danych producenta)

Parameters <i>Parametry</i>	Lacquer <i>Lakier</i>		
	A	B	C
Binding agents <i>Środki wiążące</i>	acrylic dispersion <i>dyspersja akrylowa</i>		
Solvents <i>Rozcieńczalnik</i>	water <i>woda</i>		
Content of solids [%] <i>Zawartość ciał stałych [%]</i>	32	34	38
Density [g/cm ³] <i>Gęstość [g/cm³]</i>	1.04	1.02	1.05
Commercial viscosity at a temp. of 22±1°C [s] * <i>Lepkość produktu handlowego w temp. 22±1°C [s] *</i>	30	34	47
Working viscosity at a temp. of 21±1°C [s] * <i>Lepkość produktu nanoszonego w temp. 22±1°C [s] *</i>	22	22	22

*Value measured using a Ford's cup No. 4

*Wartość mierzona za pomocą kubka Forda nr 4

The lacquers selected for investigation were applied onto samples of oak wood (*Quercus* sp.) measuring 280×200×16 mm. The moisture content of the finished wood, determined by the gravimetric method, was approximately 6.5%; while its density determined stoichiometrically in accordance with PN-77/D-04101 standard amounted to about 680 kg/m³.

The experimental samples were treated with the tested lacquers to the amount of about 110 g/m² and their narrow edges were secured by low-emission aluminium foil. The lacquer-treated wood samples were left outdoors for 24 hours which, according to the manufacturer's certificate, allowed their drying to the required level. Then, the samples were placed in a glass testing chamber of a volume of 0.225 m³. The investigations of the volatile organic compound emissions were performed in typical conditions in accordance with the recommendations of RAL-UZ 38 standard:

- temperature – $23^{\circ}\text{C} \pm 1^{\circ}\text{C}$,
- air relative humidity – $45\% \pm 1\%$,
- air exchange rate – 1 h^{-1} ,
- chamber load with examined material - $1\text{ m}^2/\text{m}^3$.

Air samples were collected 24 hours after the experimental elements were placed in the glass chamber. Each time, three parallel samples were collected as well as air that constituted the “background” of the chamber. 1000 ml of air at a rate of 50 ml/min was taken for analysis with a FLEC pump from Chematec company. The air samples were collected on Tenax TA (120mg; 35/60mesh, Alltech, Deerfield, IL).

Before application, tubes were kept at a temperature of 270°C for 60 minutes, with argon flow of 20 ml/min. Adsorbed analytes were released thermally in a desorber for 5 min. Then the liberated analytes were transferred in a stream of inert gas flowing at a rate of $20\text{ cm}^3/\text{min}$. into a microtrap. At desorption termination analytes from the microtrap were released thermally for 90 sec. at a temperature of 250°C and directed to the head of the chromatographic column. All desorbed analytes were subjected to chromatographic analysis in conditions given in table 2.

Table 2. Parameters of TD/GC/MS analytical system

Tabela 2. Parametry układu analitycznego TD/GC/MS

Elements of the system <i>Elementy układu</i>	Parameters <i>Parametry</i>
Gas chromatograph <i>Chromatograf gazowy</i>	TRACE GC, Thermo Quest.
Column <i>Kolumna</i>	RTX – 624 Restek Corporation, 60m x 0,32mm ID; $D_f - 1,8\ \mu\text{m}$: 6% cyanopropylophenyl, 94% dimetylopolisiloxane RTX – 624 Restek Corporation, 60m x 0,32mm ID; $D_f - 1,8\ \mu\text{m}$: 6% cyjanopropylofenyl, 94% dimetylopolisiloksan
Detector <i>Detektor</i>	Mass spectrometer (SCAN: 10 – 350) <i>Spektrometr mas (SCAN: 10 – 350)</i>
Injector <i>Dozownik</i>	Thermal desorber connected with sorption microtrap; Rinsing gas: argon $20\text{ m}^3\text{min}^{-1}$; Rinsing time: 5 min. <i>Termiczny desorber połączony z pułapką sorbcyjną;</i> <i>Gaz płuczący: argon $20\text{ m}^3\text{min}^{-1}$;</i> <i>Czas płukania: 5 min.</i>
Microtrap <i>Mikropułapka</i>	Sorbent: 80 mg Tenax TA/30 mg Carbosieve III; Desorption temperature: 250°C during 90 s. <i>Sorbent: 80 mg Tenax TA/30 mg Carbosieve III;</i> <i>Temperatura desorpcji: 250°C przez 90 s.</i>
Carrier gas <i>Gaz nośny</i>	Helium: 100 kPa, $\sim 2\text{ cm}^3\text{min}^{-1}$. <i>Hel: 100 kPa, $\sim 2\text{ cm}^3\text{min}^{-1}$.</i>

Table 2. Continued
Tabela 2. Ciąg dalszy

Temperature setting <i>Program temperaturowy</i>	40°C during 2 min, 7°C min ⁻¹ to 200°C, 10°C min ⁻¹ to 230°C, 230°C during 20 min <i>40°C przez 2min, 7°C min⁻¹ do 200°C, 10°C min⁻¹ do 230°C, 230°C przez 20 min.</i>
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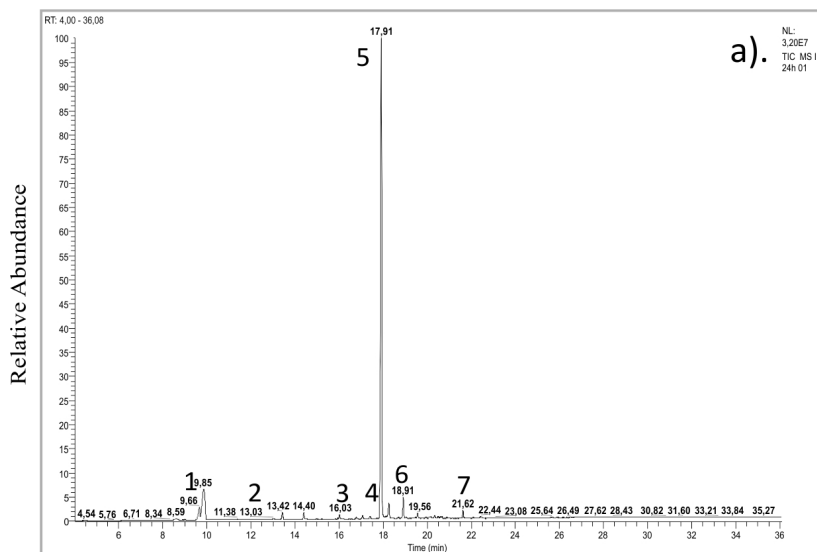
Compound identification: compounds were identified by comparing the mass spectra obtained with the spectra stored in NIST 98 library and then confirmed by collating the mass spectra and retention times of the identified compounds with the spectra and retention times of appropriate standards.

Quantitative analysis: a quantitative analysis of the volatile organic compounds emitted from the examined surfaces was carried out using the method of addition of 4-bromo-1-fluorobenzene standard.

In order to determine change trends in the emission of volatile compounds in time, the investigations were carried out in cycles: 24 hours, 14 days, and 28 days after finishing the wood samples with the tested lacquers.

Results and discussion

Measurements of the concentrations of harmful substances present in the air collected from the chamber filled with the examined materials revealed quantitative and qualitative differences in the volatile organic compounds emitted by those materials. Fig. 1 presents a comparison of the profiles of the volatile organic compounds emitted from the samples coated with waterborne lacquers.



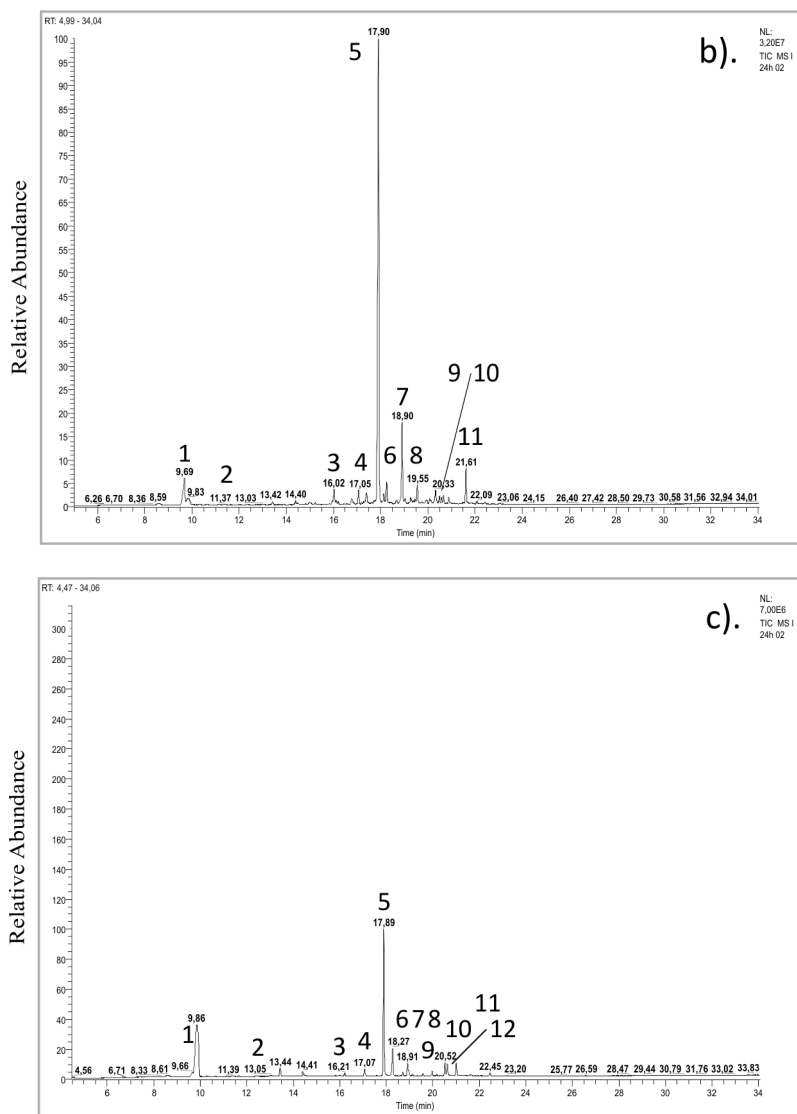


Fig. 1. Chromatograms of separation of volatile organic compounds emitted by oak wood surfaces coated with: a) lacquer A, b) lacquer B, c) lacquer C, 24 hours after the treatment

Rys. 1. Chromatogramy rozdzielu lotnych związków organicznych emitowanych przez powierzchnie drewna dębowego pokryte: a) lakierem A, b) lakierem B, c) lakierem C, po 24h od ich uszlachetnienia

The results obtained indicate that, after application onto the surface of oak wood, the investigated waterborne lacquers primarily emitted compounds from the group of glycol ethers, and aliphatic and aromatic hydrocarbons. The domi-

nant group of compounds released by all the tested products comprised glycol ethers. Their proportion of the total emission during the first stage of experiments ranged from 64.5 to 85.3%. In addition, wood surfaces finished with lacquers A and B also released relatively high quantities of aliphatic hydrocarbons (from 5.5 to 21.5%); while samples coated with lacquer C emitted significant amounts of aromatic hydrocarbons (15.4%).

Tables 3-5 present detailed results of measurements of volatile organic compounds identified in the air collected from the chamber filled with the tested materials. The results present the mean concentration values of the volatile compounds from three measurements. The values did not differ from one another by more than 10%.

Table 3. Volatile organic compounds released from coatings of waterborne lacquer A applied onto oak wood

Tabela 3. Lotne związki organiczne wydzielane z powłok wodorozcieńczalnego lakieru A naniesionych na drewno dębowe

Compound <i>Związek</i>	Compound designation <i>Oznaczenie związku</i>	Compound concentration <i>Stężenie związków</i>		
		24 hours <i>24h</i>	14 days <i>14 doba</i>	28 days <i>28 doba</i>
		µg/m ³		
acetic acid <i>kwask octowy</i>	1	49.8	10.1	6.2
toluene <i>toluen</i>	2	5.0	3.9	1.7
n-nonane <i>n-nonan</i>	3	10.0	4.2	3.8
cyclohexane, propyl <i>propylocykloheksan</i>	4	8.2	1.7	1.4
ethanol, 2-butoxy <i>2-butoksyetanol</i>	5	1531.0	415.4	137.3
n-decane <i>n-dekan</i>	6	60.2	6.9	4.9
n-undecane <i>n-undekan</i>	7	20.7	1.8	1.0
Σ unidentified substances <i>Σ niezidentyfikowanych substancji</i>	-	109.1	24.1	22.7
TVOC ¹ :		1794	468	179

¹Total emission of volatile organic compounds

¹*Całkowita emisja lotnych związków organicznych*

- Undetected

- *Nie stwierdzono*

Table 4. Volatile organic compounds released from coatings of waterborne lacquer B applied onto oak wood**Tabela 4. Lotne związki organiczne wydzielane z powłok wodorozcieńzalnego lakieru B naniesionych na drewno dębowe**

Compound <i>Związek</i>	Compound designation <i>Oznaczenie związku</i>	Compound concentration <i>Stężenie związków</i>		
		24 hours <i>24h</i>	14 days <i>14 doba</i>	28 days <i>28 doba</i>
		$\mu\text{g}/\text{m}^3$		
acetic acid <i>kwask octowy</i>	1	71.5	15.7	9.7
toluene <i>toluen</i>	2	4.7	4.4	3.4
n-nonane <i>n-nonan</i>	3	37.8	10.5	6.2
cyclohexane, propyl <i>propylocykloheksan</i>	4	29.1	6.1	4.1
ethanol, 2-butoxy <i>2-butoksyetanol</i>	5	1086.9	517.4	251.7
nonane, 3-methyl <i>3-metylononan</i>	6	12.7	4.3	2.3
n-decane <i>n-dekan</i>	8	149.4	31.3	22.1
nonane, 2,6-dimethyl <i>2,6-dimetylononan</i>	9	31.7	10.5	6.7
cyclohexane, butyl <i>butylocykloheksan</i>	10	26.7	6.4	5.5
decane, 3-methyl <i>3-metylodekan</i>	11	23.2	14.7	8.6
n-undecane <i>n-undekan</i>	12	52.1	12.7	4.8
Σ unidentified substances <i>Σ niezidentyfikowanych substancji</i>	-	158.2	63.0	44.9
TVOC ¹ :		1684	697	370

¹Total emission of volatile organic compounds¹Calkowita emisja lotnych związków organicznych

- Undetected

- Nie stwierdzono

Table 5. Volatile organic compounds released from coatings of waterborne lacquer C applied onto oak wood**Tabela 5. Lotne związki organiczne wydzielane z powłok wodorozcieńczalnego lakieru C naniesionych na drewno dębowe**

Compound <i>Związek</i>	Compound designation <i>Oznaczenie związku</i>	Compound concentration <i>Stężenie związków</i>		
		24 hours <i>24h</i>	14 days <i>14 doba</i>	28 days <i>28 doba</i>
		$\mu\text{g}/\text{m}^3$		
acetic acid <i>kwias octowy</i>	1	12.7	5.2	5.1
toluene <i>toluene</i>	2	3.8	2.4	1.8
m-xylene <i>m-ksylen</i>	3	5.3	3.1	1.0
o-xylene <i>o-ksylen</i>	4	13.1	7.6	-
ethanol. 2-butoxy <i>2-butoksyetanol</i>	5	231.7	46.1	13.9
benzene. propyl <i>propylobenzen</i>	6	3.9	2.9	2.4
1.2.3-trimethylbenzene. 1.2.3-trimetylobenzen	7	26.4	12.1	-
1-ethyl-4-methylbenzene 1-etylo-4-metylobenzen	8			
1.3.5-trimethylbenzene. 1.3.5-trimetylobenzen	9	7.3	2.9	-
1-(2-methoxy-1-methylethoxy)- -2-propanol <i>1-(2-metoksy-1-metyloetoksy)-2-propanol</i>	10	19.7	12.8	11.9
dipropylene glycol methyl ether (isomer) <i>eter metylowy glikolu dipropylenowego (izomer)</i>	11	19.6	14.9	13.9
1-(2-methoxypropoxy)-2-propanol <i>1-(2-metoksypropoksy)-2-propanol</i>	12	20.4	18.6	17.6
Σ unidentified substances <i>Σ niezidentyfikowanych substancji</i>	-	24.1	17.4	16.9
TVOC ¹ :		388	146	85

¹Total emission of volatile organic compounds¹Calkowita emisja lotnych związków organicznych

- Undetected

- Nie stwierdzono

The main constituent released by all examined materials was 2-butoxyethanol. The concentration of that compound ranged from 231.7 to 1531.0 $\mu\text{g}/\text{m}^3$. Among the tested lacquers, the lacquer coatings containing the highest amounts of solids (38%, lacquer C) emitted the smallest quantities of 2-butoxyethanol; while the highest quantities of that compound were released by surfaces finished with lacquer A, which contained the smallest amounts of solids (32%).

The surfaces finished with lacquer C also emitted other compounds belonging to glycol ethers, e.g. 1-(2-methoxy-1-methylethoxy)-2-propanol, methyl ether of dipropylene glycol, as well as 1-(2-methoxypropoxy)-2-propanol. Characteristic components of emissions from coatings of A and B lacquers also included compounds belonging to aliphatic hydrocarbons. Coatings of lacquer A released primarily: n-nonane, n-decane, n-undecane; whereas coatings of lacquer B also emitted propylcyclohexane, butylcyclohexane, 3-methylnonane, 2,6-dimethylnonane or 3-methyldecane. On the other hand, surfaces finished with lacquer C released aromatic hydrocarbons of which the most interesting were emissions of m-,o-xylene as well as 1,2,3-trimethylbenzene and 1,3,5-trimethylbenzene.

All examined samples also emitted acetic acid to the amount of 12.7-71.5 $\mu\text{g}/\text{m}^3$. The source of acetic acid emissions was oak wood, which was confirmed by experiments carried out on untreated oak wood samples as well as investigations conducted by Risholm-Sundman et al. [1998].

The total concentrations of all volatile organic compounds released by examined surfaces 24 hours after the treatment are presented in fig. 2. The overall quantity of compounds emitted by coatings of waterborne lacquers ranged from 388 to 1794 $\mu\text{g}/\text{m}^3$. The greatest quantities of volatile organic compounds were released by coatings of lacquer A, which contained the lowest amounts of solids. In the first stage of the experiments, the amount of compounds liberated from coatings developed from lacquer A was over 4.5 fold higher than that determined for coatings formed by lacquer C, characterised by the highest content of solids. On the other hand, the total quantity of all volatile compounds released by surfaces finished with A and B lacquers 24 hours after the treatment differed only slightly. However, the profile of compounds released into the atmosphere was significantly more diverse.

During the first 14 days the emission levels of all identified compounds decreased significantly. For example, in the case of surfaces finished with lacquer A, concentrations of 2-butoxyethanol, which constituted the main compound released by waterborne coatings, decreased by approximately 73%, in the case of lacquer B by about 52% and in the case of lacquer C by about 80%.

The quantity of all compounds (TVOC) emitted by waterborne coatings in the course of 14 days decreased by about 59 to 74%. Fourteen days after the treatment, experimental materials released volatile organic compounds at the level of 146-697 $\mu\text{g}/\text{m}^3$. The greatest drop of emissions was recorded for product A characterised by the highest emissions in 24 hours after lacquer application; while the lowest was observed in the case of product B.

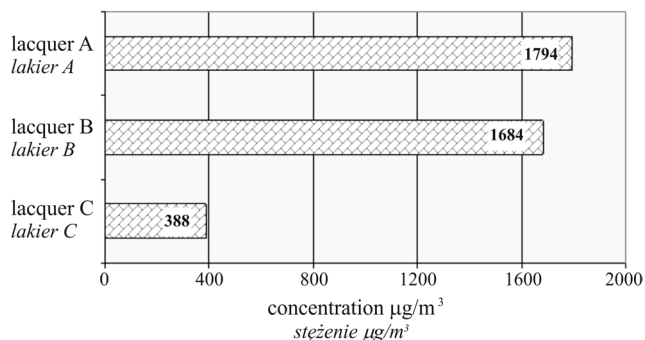


Fig. 2. Comparison of TVOC released by coatings of waterborne lacquers 24 hours after their application onto the surface of oak wood

Rys. 2. Porównanie TVOC wydzielanych przez powłoki wodorozcieńczalne po 24h od nanieśienia ich na powierzchnię drewna dębu

In the course of the following 14 days, quantities of volatile organic compounds released by coatings of waterborne lacquers decreased further, ranging from 85- 370 µg/m³. The lowest emissions of volatile organic compounds 28 days after the treatment were determined in the case of product C and the highest for product B, similar to the results recorded 14 days after application of tested lacquers.

To summarise, it can be said that the quantities of volatile organic compounds released by the examined coatings of waterborne lacquers during the first 28 days after their application onto oak wood surfaces decreased by 78 to 90%.

The quantitative changes of VOC released by the coatings of waterborne lacquers during the first 28 days after their application onto oak wood are given in fig. 3.

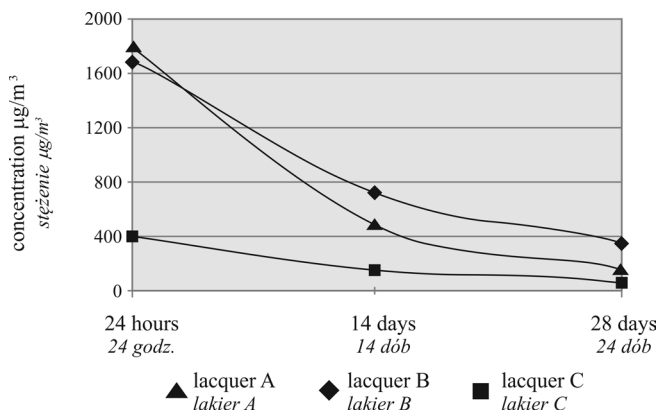


Fig. 3. Change of volatile organic compounds concentrations in time for examined coatings applied onto oak wood

Rys. 3. Zmiany stężenia lotnych związków organicznych w czasie, wydzielanych przez badane powłoki naniesione na drewno dębu

Conclusions

The conducted research confirmed the need for qualitative and quantitative characterisation of harmful substances emitted by surfaces finished with acrylic waterborne lacquers. Moreover, it also confirmed the fact that emission levels of volatile organic compounds released from these products may change in a wide range of values. In the case of the two tested products (lacquer A and B), the total content of all volatile organic compounds released into the air collected from the chamber was relatively high and, 24 hours after their application onto the surface of oak wood, fluctuated from 1794 to 1684 $\mu\text{g}/\text{m}^3$. Much lower emissions of volatile organic compounds were determined in the case of coatings of lacquer C (388 $\mu\text{g}/\text{m}^3$) characterised by the highest content of solids of all tested products.

The highest quantities of compounds released by examined lacquers belonged to glycol ethers. The principal constituent of those emissions was 2-butoxyethanol, which constituted from 59.7 to 81.1% of all released compounds. In addition, those emissions also contained small quantities of aliphatic and aromatic hydrocarbons. It is worth mentioning that aliphatic hydrocarbons were characteristic constituents of emissions from coatings developed from A and B lacquers; whereas aromatic hydrocarbons were released in significant amounts from coatings formed by lacquer C.

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EMISJA LOTNYCH ZWIĄZKÓW ORGANICZNYCH (VOC) Z WODOROZCIEŃCZALNYCH WYROBÓW LAKIEROWYCH O ZRÓŻNICOWANEJ ZAWARTOŚCI CIAŁ STAŁYCH

Streszczenie

Przedstawiono wyniki badań emisji lotnych związków organicznych z powierzchni drewna dębowego, uszlachetnionego akrylowymi lakierami wodorozcieńczalnymi. Użyte wyroby lakierowe charakteryzowały się różną zawartością ciał stałych, która zmieniała się w zakresie od 32 do 38%. Lotne związki organiczne analizowano techniką chromatografii gazowej połączonej ze spektrometrią mas i termiczną desorpcją (GC/MS/TD). Jako medium adsorpcyjne zastosowano Tenax TA. Uzyskane rezultaty wskazują, iż emisja lotnych związków organicznych z wyrobów wodorozcieńczalnych zmieniała się w szerokim zakresie, od 388 do 1794 $\mu\text{g}/\text{m}^3$. Głównymi składnikami emisji były związki należące w większości do eteroglikoli, węglowodorów alifatycznych oraz aromatycznych.

Słowa kluczowe: lakiery wodorozcieńczalne, lotne związki organiczne (VOC), chromatografia gazowa, spektrometria mas, termiczna desorpcja